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Letters to the Editor

X-ray crystallographic data on homophthalic acid

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As part of a programme of determining the crystal structure of simple organic molecules, we have examined homophthalic acid (α-2-toluene dicarboxylic acid), $C_6H_4(CH_2COOH)\cdot COOH$, using X-ray diffraction techniques.

Colourles, platy crystals of homophthalic acid were grown out of water solution. The platy face is designated as (010) and the bounding edges as [101], [001] and [100]. The angles between [100] and [101] is $\sim 37°$ and between [001] and [100] $\sim 65°$. In some crystals only the [101] and [100] directions appear as the bounding edges of the face [010], the crystal resembling an elongated rhombus.

Examined under the polarizing microscope, with the [010] face horizontal, the extinction direction makes an angle of $\sim 12°$ with the [100] axis of the crystal. Laue photographs with [010] face perpendicular to the X-ray beam showed two well defined zone directions which were taken to be [001] and [100]. The average value of the measurements on these laue photographs gave β or $(180-\beta)$ as 115°35′. Rotation photographs along [001] and [100] gave the identity periods to be 5.23 Å and 8.45 Å. Normal beam Weissenberg zero layer photographs along [00] and [100] gave A or $180°-\alpha^*$ and C or $180° - \gamma^*$ as 97.4° and 92° respectively. The values of A, C and β or $180 - \beta$ gave rise to two possibilities for the interaxial angles α,β and γ i. e.

$$\alpha = 80°54' \quad \beta = 115°35' , \qquad \gamma = 95°45' \qquad \ldots (1)$$

$$\text{and } \alpha = 82°46' \quad \beta = 64°25' \qquad \gamma = 88°40' \qquad \ldots (2)$$

Laue and oscillation photographs along the perpendicular directions of the (100) and (008) faces gave evidence of the first possibility of the angles α, β and γ.

Finally from the combined evidence of normal beam zero layer Weissenberg photographs along [010], [100], [001], [101] and [10$\bar{1}$] axes, rotation photographs along [100], [001], [021] and [012] axes and Laue photographs along [001], [100] and [010] axes the following unit cell constant were obtained, i. e.

$a = 8.45 \pm 0.02$ $\quad \alpha = 80°3'$
$b = 10.87 \pm 0.03$ $\quad \beta = 116°10'$
$c = 5.23 \pm 0.01$ $\quad \gamma = 96°44'$

which tallies with the first possibility of the angles α, β and γ. Index powder data using the above unit cell constants for homophthalic acid will be found in table 1 below. Powder photograph with the powered sample sealed in Lindemann glass capillary with internal diameter 0.3 mm, was taken in a 9 cm. unicam powder camera with Van-Arkel arrangement. Copper (Ni-filtered) radiation was used for photographing the powder lines.

TABLE 1

I/I_0 Relative Intensity.	Index h k l	d obs (A°)	d cal (A°)	I/I_0 Relative Intensity	Index h k l	d obs (A°)	d cal (A°)
64	110	6.08	6.05	4	$3\bar{1}0$ }	2.47	2.48 }
2	020	5.36	5.35		041 }		2.47 }
100	$10\bar{1}$	5.10	5.12		$\bar{1}22$ }		2.47 }
4	001	4:70	4.65	1	212	2.36	2.36
36	011	4.50	4.49	5	221	2.28	2.27
45	120 }	4.27	4 28 }	2	022 }	2.25	2.25 }
	111 }		4.28 }		320 }		2.24 }
73	$01\bar{1}$	4.08	4.07	3	$12\bar{2}$	2.18	2.17
13	200 }	3.79	3.79 }	1	240 }	2.12	2.14 }
	$02\bar{1}$ }		3.78 }		$3\bar{3}0$ }		2.11 }
6	210	3.53	3.52	1	032	2.09	2.08
3	$\bar{1}21$	3.38	3.38	3	$0\bar{2}2$	2.04	2.03
73	$02\bar{1}$ }	3 .29	3.29 }	3	$11\bar{2}$	1.92	1.92
	111 }		3.29 }	2	032	1.84	1.84
	$\bar{1}30$ }		3.28 }	2	340 }	1.79	1.80 }
8	$2\bar{2}0$	3.16	3.16		060 }		1.78 }
13	031	3.05	3.04	2	$\bar{1}60$	1.75	1.75
2	$22\bar{1}$	2.91	2.92	3	$\bar{1}13$	1.69	1.69
5	301 }	2.77	2.77 }	4	042 }	1.65	1.65 }
	$\bar{3}11$ }		2.78 }		$2\bar{1}2$ }		1.65 }
					222 }		1.64 }
8	$03\bar{1}$ }	2.66	2.66 }	V.V.W.	440 }	1.51	1.51 }
	$\bar{2}30$ }		2.66 }		500 }		1.52 }
11	230	2.54	2.54	V.V.W.	302 }	1.43	1.43 }
					043 }		1.43 }

The density of the crystal measured by floatation method was 1.41 gm/cc and that calculated with 2 molecules per unit cell was 1.423 gms/cc.

Evidences from the morphology of the crystal, intensity, statistics (Howells *et al* 1950) as well as the number of molecules per unit cell suggest the space group to be P $\bar{1}$. Pyroelectric tests on the crystal gave negative results.

A full investigation of the crystal structure using Fourier techniques and partial thee-dimensional data is in progress, the details of which will be published later.

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REFERENCE

Howells, E. R. Phillips, D. C. Rogers, D. 1950, Acta Cryst. 3, 210.

On the co-efficient of molecular packing in some dicarboxylic acids.

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Kitaigorodski (1961) has put forward the theory that "a crystal of an organic compound can be considered as a system of closely packed layers, the molecules within a layer are also very closely packed, having co-ordination numbers of six and being so arranged that no polarities appear perpendicular to the layer". He has also suggested that "the intermolecular radii elements found in organic compounds are universal and reasonably constant *i. e.* applicable to all molecules. They can be used to assign definite shapes to molecules. If, after the molecules have been built up from intermolecular radii, we consider the molecular posi-